

DRUG CHEMISTRY SECTION TECHNICAL PROCEDURE MANUAL		
Procedure A-01	Preliminary Tests Marquis Reagent	
Effective Date:	April 15, 2010	Page 1 of 3

**Name of Procedure:**

Preliminary Tests  
Marquis Reagent

**Suggested Uses:**

The Marquis reagent consists of a solution of formaldehyde in concentrated sulfuric acid. Aromatic compounds that typically undergo electrophilic substitution will react with the Marquis reagent to produce colored intermediates. A positive response with the Marquis reagent is indicated by a significant color formation within 1-2 minutes. Refer to pages 139-140, **Clarke's Isolation and Identification of Drugs**, and pages 631-649, "Spot Tests: A Color Chart Reference for Forensic Chemists", (see **Literature References**) for color formations of various drugs.

**Apparatus Needed to Perform Procedure Including Preparation of Reagent:**

Fume hood  
Gloves  
Eye protection  
Laboratory coat  
Pipet with bulb  
Graduated cylinder  
50ml beaker  
Glass stirring rod  
Sulfuric acid (concentrated)  
Formaldehyde solution (40%)  
Funnel  
Reagent bottle  
Porcelain spot plate  
Spatula

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### **Formula for Preparing Reagent:**

1. Measure out 10 milliliters of concentrated sulfuric acid in a beaker.
2. Add 8-10 drops of formaldehyde solution (40%) and stir.
3. Pour solution into a reagent bottle.
4. Properly label reagent bottle.

### **Alternate Method**

1. Pour 15-20 milliliters of concentrated sulfuric acid into a reagent bottle.
2. Add 0.2 - 0.3 gram of trioxane (trioxymethylene) and stir until completely dissolved.
3. Properly label reagent bottle.

### **Quality Control Check:**

A quality control check of this reagent will be performed using a known standard of guaifenesin and following the application procedure listed below.

### **Expiration Date of Chemical Reagent:**

The Marquis reagent should be prepared every six months if stored in a refrigerator or every 60 days if store on the bench top.

### **Application of Procedure on Evidence:**

1. Place 1-2 drops of the reagent into a clean well on a spot plate.
2. With a spatula, add approximately 0.1 milligram of the unknown powder/tablet to the reagent in the spot plate.

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3. Observe color produced.
4. Record results.

### **Safety Concerns:**

Always wear eye protection, gloves and a laboratory coat when preparing this reagent.

Eye protection and a laboratory coat should be worn when using this reagent for color tests.

Sulfuric acid is a strong oxidizing agent and corrosive.

### **Literature References:**

Moffat, A. C., ed., **Clarke's Isolation and Identification of Drugs**, 2nd Ed., Pharmaceutical Press, London, 1986, p. 139-140..

Johns, S. H., "Spot Tests: A Color Chart Reference for Forensic Chemists," **Journal of Forensic Science**, July, 1979, pp. 631-649.

Butler, William P., **Methods of Analysis**, IRS Publication #341, December 1966, p. 136.

This procedure has been used in the Drug Chemistry Section since 1971.

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Procedure A-02	Preliminary Tests Duquenois-Levine (Modified)	
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**Name of Procedure:**

Preliminary Tests  
Duquenois-Levine Reagent (Modified)

**Suggested Uses:**

The Duquenois-Levine reagent is used in the identification of marijuana. If cannabinoids are present, an intense violet blue color develops. When shaken with chloroform, the color is transferred to the chloroform phase.

**Apparatus Needed to Perform Procedure Including Preparation of Reagent:**

Fume hood  
Gloves  
Eye protection  
Laboratory coat  
Pipet with bulb  
Graduated cylinder  
(2) 30ml reagent bottles  
Glass stirring rod  
6 X 50mm culture tubes  
250ml beaker  
Acetaldehyde  
Vanillin  
Ethanol  
Chloroform  
Hydrochloric acid  
Funnel  
Reagent bottle with dropper  
Spatula  
Porcelain spot plate

**Formula for Preparing Reagent:**

1. Measure out 2.5 milliliters acetaldehyde

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2. Measure out 2.0 grams vanillin.
3. Measure out 100 milliliters of ethanol.
4. Dissolve vanillin and acetaldehyde in ethanol in 250 milliliter beaker.
5. Pour into reagent bottle.
6. Properly label reagent bottle.
7. Store in dark place.
8. Fill separate reagent bottle with chloroform and properly label.

**Quality Control Check:**

A quality control check of this reagent will be performed using a known standard of marijuana and following the application procedure listed below.

**Expiration Date of Chemical Reagent:**

No expiration date if the reagent is stored in a sealed container in a refrigerator (stock solution). If the reagent is not stored in the refrigerator, it should be replaced every three months.

**Application of Procedure on Evidence:**

1. Place approximately 1 milligram of sample material in a culture tube or spot plate.
2. Add 2-3 drops of the Duquenois reagent.
3. Add 4-5 drops of concentrated hydrochloric acid and observe the color changes.
4. Add 2-3 drops of chloroform and agitate.

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5. Allow phases to separate and observe the color in the bottom chloroform layer.  
A blue to violet color with the acid addition, a violet color transfer to the chloroform layer is indicative of a positive test.
6. Record results.

#### **Safety Concerns:**

Always wear eye protection, gloves and a laboratory coat when preparing this reagent.

Eye protection and a laboratory coat should be worn when using this reagent for color tests.

#### **Literature References:**

Moffat, A. C. ed., **Clarke's Isolation and Identification of Drugs**, 2nd Ed., Pharmaceutical Press, London, 1986, p. 133.

Butler, William P., **Methods of Analysis**, IRS Publication #341, December 1966, p. 105.

This procedure has been used in the Drug Chemistry Section since 1971.

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Procedure A-03	Preliminary Tests Cobalt Thiocyanate Reagent	
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**Name of Procedure:**

Preliminary Tests  
Cobalt Thiocyanate Reagent

**Suggested Uses:**

The Cobalt Thiocyanate reagent consists of an aqueous solution of Cobalt thiocyanate. This reagent reacts with some secondary and tertiary amines and other alkaloids to produce a blue color.

**Apparatus Needed to Perform Procedure Including Preparation of Reagent:**

Fume hood  
Gloves  
Eye protection  
Laboratory coat  
Pipet with bulb  
Graduated cylinder  
50ml beaker  
Cobaltous thiocyanate  
Water  
Glass stirring rod  
Funnel  
Reagent bottle  
Porcelain spot plate  
Spatula

**Formula for Preparing Reagent:**

1. Measure out 2.0 grams of Cobaltous thiocyanate.
2. Dissolve in 100 milliliters of water.
3. Pour into a reagent bottle.

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4. Properly label reagent bottle.

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### **Formula for Preparing Reagent (continued):**

#### **Alternate Method:**

1. Weigh out 0.4 gram of ammonium thiocyanate.
2. Add ammonium thiocyanate to 25 milliliters of water and dissolve.
3. Weigh out 0.7 gram of cobalt acetate.
4. Dissolve the cobalt acetate in the solution of ammonium thiocyanate.
5. Pour into a reagent bottle.
6. Properly label reagent bottle.

#### **Quality Control Check:**

A quality control check of this reagent will be performed using a known standard of cocaine and following the application procedure listed below.

#### **Expiration Date of Chemical Reagent:**

No expiration date. Reagents need to be properly contained in a sealed container and stored in a cool place.

#### **Application of Procedure on Evidence:**

1. Place 1-2 drops of the reagent into a clean well of a spot plate.
2. With a spatula, add approximately 0.1 milligram of the unknown powder/tablet to the reagent in the spot plate.
3. Observe the color produced.

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4. Record results.

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### **Safety Concerns:**

Always wear eye protection and laboratory coat when preparing this reagent. A laboratory coat should be worn when using this reagent for color tests.

### **Literature References:**

Butler, William P., **Methods of Analysis**, IRS publication #341, December 1966, p.136.

Johns, S.H., "Spot Tests: A Color Chart Reference for Forensic Chemists", **Journal of Forensic Science**, July, 1979, pp. 631-649.

This procedure has been used in the Drug Chemistry Section since 1971.

DRUG CHEMISTRY SECTION TECHNICAL PROCEDURE MANUAL		
Procedure A-04	Preliminary Tests Ferric Chloride Reagent	
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**Name of Procedure:**

Preliminary Tests  
Ferric Chloride Reagent

**Suggested Uses:**

The ferric chloride reagent consists of a solution of ferric chloride in water. This reagent reacts with phenols, enols and other functional groups to give colored solutions in less than 30 seconds. Refer to page 133, **Clarke's Isolation and Identification of Drugs** (see **Literature References**) for color formations of various drugs.

**Apparatus Needed to Perform Procedure Including Preparation of Reagent:**

Fume hood  
Gloves  
Eye protection  
Laboratory coat  
Pipet with bulb  
Graduated cylinder  
50ml beaker  
Glass stirring rod  
Funnel  
Reagent bottle  
Porcelain spot plate  
Spatula  
Ferric chloride  
Water

**Formula for Preparing Reagent:**

1. Measure out 1.5 grams of ferric chloride.
2. Dissolve ferric chloride in 29.0 milliliters of water.
3. Pour solution into a reagent bottle.

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4. Properly label reagent bottle.

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### **Quality Control Check:**

A quality control check of this reagent will be performed using a known standard of acetaminophen and following the application procedure listed below.

### **Expiration Date of Chemical Reagent:**

No expiration date. Reagents need to be properly contained in a sealed container and stored in a cool place.

### **Application of Procedure on Evidence:**

1. Place 1-2 drops of the reagent into a clean well of a spot plate.
2. With a spatula, add approximately 0.1 milligram of the unknown powder/tablet to the reagent in the spot plate.
3. Observe the color produced.
4. Record results.

### **Safety Concerns:**

Always wear eye protection and laboratory coat when preparing this reagent. A laboratory coat should be worn when using this reagent for color tests.

### **Literature References:**

Moffat, A. C., ed., **Clarke's Isolation and Identification of Drugs**, 2nd Ed., Pharmaceutical Press, London, 1986, p. 133.

This procedure has been used in the Drug Chemistry Section since 1971.

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Procedure A-05	Preliminary Tests Koppanyi Reagent	
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**Name of Procedure:**

Preliminary Tests  
Koppanyi Reagent

**Suggested Uses:**

The Koppanyi reagent consists of filter paper treated with (soaked in) cobalt acetate, methanol and glacial acetic acid and a solution of 5% isopropylamine in methanol. Barbiturates (5,5-disubstituted malonylureas) and other compounds will produce a red-violet color formation in less than 30 seconds. Refer to pages 631-649, "Spot Tests: A Color Chart Reference for Forensic Chemists", (see **Literature References**) for color formations with various other drugs.

**Apparatus Needed to Perform Procedure Including Preparation of Reagent:**

Fume hood  
Gloves  
Eye protection  
Laboratory coat  
Pipet with bulb  
Graduated cylinder  
50ml beaker  
Glass stirring rod  
Filter paper  
Scissors  
Glacial acetic acid  
Cobalt acetate  
Methanol  
Wide mouth bottle with top  
Funnel  
Reagent bottle  
Porcelain spot plate  
Spatula

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### **Formula for Preparing Reagent:**

1. Dissolve 0.1 gram of cobalt acetate in 100 milliliters of methanol.
2. Add 0.2 milliliter of glacial acetic acid.
3. Soak filter paper in the solution and allow to completely dry.
4. Cut filter paper into approximately 1 inch squares.
5. Store filter paper in a wide mouth bottle with top.
6. Make a separate solution of 5% isopropylamine in methanol.
7. Pour into reagent bottle.
8. Properly label reagent bottles.

### **Quality Control Check:**

A quality control check of this reagent will be performed using a known standard of a barbiturate and following the application procedure listed below.

### **Expiration Date of Chemical Reagent:**

No expiration date. Reagents need to be properly contained in a sealed container and stored in a cool place.

### **Application of Procedure on Evidence:**

1. Place a small amount (approximately 0.1 milligram) of sample on a piece of the Koppanyi paper.



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2. Press the sample into the paper well with a spatula.
3. Place a drop of 5% isopropylamine solution on the edge of the Koppanyi paper and allow the drop to meet the sample.
4. Observe the color produced.
5. Record results.

**Safety Concerns:**

Always wear eye protection, gloves and a laboratory coat when preparing this reagent.

Eye protection and a laboratory coat should be worn when using this reagent for color tests.

**Literature References:**

Butler, William P., **Methods of Analysis**, IRS publication #341, December 1966, p.106.

Johns, S.H., "Spot Tests: A Color Chart Reference for Forensic Chemists", **Journal of Forensic Science**, July, 1979, pp. 631-649.

This procedure has been used in the Drug Chemistry Section since 1971.

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Procedure A-06	Preliminary Tests Potassium Permanganate Reagent	
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**Name of Procedure:**

Preliminary Tests  
Potassium Permanganate Reagent

**Suggested Uses:**

The Potassium Permanganate reagent consists of a solution (purple) of potassium permanganate in water. Compounds containing reactive double bonds and other functional groups react to form a brown color.

**Apparatus Needed to Perform Procedure Including Preparation of Reagent:**

Fume hood  
Gloves  
Eye protection  
Laboratory coat  
Pipet with bulb  
Graduated cylinder  
50ml beaker  
Glass stirring rod  
Potassium permanganate  
Funnel  
Reagent bottle  
Porcelain spot plate  
Spatula  
Water  
Culture tube (6 X 50mm)

**Formula for Preparing Reagent:**

1. Weigh out 0.3 gram of potassium permanganate.
2. Dissolve in 30 milliliters water.
3. Pour into a reagent bottle.

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4. Properly label reagent bottle.

#### **Quality Control Check:**

A quality control check of this reagent will be performed using a known standard of an opiate and following the application procedure listed below.

#### **Expiration Date of Chemical Reagent:**

No expiration date. Reagents need to be properly contained in a sealed container and stored in a cool place.

#### **Application of Procedure on Evidence:**

1. Place approximately 0.1 milligram sample in a culture tube (6 X 50mm) with a spatula.
2. Add 1-2 drops of potassium permanganate solution.
3. Observe for discoloration of potassium permanganate reagent color.
4. Record results.

#### **Safety Concerns:**

Always wear eye protection and laboratory coat when preparing this reagent. A laboratory coat should be worn when using this reagent for color tests.

#### **Literature References:**

Moffat, A. C., ed., **Clarke's Isolation and Identification of Drugs**, 2nd Edition, Pharmaceutical Press, London, 1986, p. 1170.

This procedure has been used in the Drug Chemistry Section since 1971.

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Procedure A-07	Preliminary Tests p-Dimethylaminobenzaldehyde Reagent (PDMAB)	
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**Name of Procedure:**

Preliminary Tests  
p-Dimethylaminobenzaldehyde Reagent (PDMAB)

**Suggested Uses:**

The PDMAB color reagent consists of filter paper soaked in a PDMAB/methanol solution and dried. Indoles react to produce a purple color formation in less than 30 seconds. Primary aromatic amines undergo catalyzed condensation reactions with PDMAB to form yellow-orange imine compounds. Carbamates also react with the PDMAB reagent. Refer to page 132, **Clarke's Isolation and Identification of Drugs**, and pages 631-649, "Spot Tests: A Color Reference for Forensic Chemists", (see **Literature References**) for color formations of various drugs.

**Apparatus Needed to Perform Procedure Including Preparation of Reagent:**

Fume hood  
Gloves  
Eye protection  
Laboratory coat  
Pipet with bulb  
Graduated cylinder  
50ml beaker  
Glass stirring rod  
p-Dimethylaminobenzaldehyde  
Methanol  
Filter paper  
Wide mouth bottle with top  
Hydrochloric acid  
Funnel  
Reagent bottle  
Porcelain spot plate  
Spatula

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### **Formula for Preparing Reagent:**

1. Weigh out 1.0 gram of PDMAB.
2. Dissolve in 100 milliliters of methanol.
3. Pour into a large beaker or porcelain pan.
4. Soak the filter paper in the solution.
5. Remove the filter paper and allow to dry completely.
6. Cut filter paper into 1 inch squares (approximate).
7. Store PDMAB paper in a wide mouth bottle with top.
8. Pour concentrated hydrochloric acid into a separate reagent bottle.
9. Pour methanol into a separate reagent bottle.
10. Properly label reagent bottles.

### **Quality Control Check:**

A quality control check of this reagent will be performed using a known standard of LSD and following the application procedure listed below.

### **Expiration Date of Chemical Reagent:**

No expiration date. Reagents need to be properly contained in a sealed container and stored in a cool place.

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### **Application of Procedure on Evidence:**

1. Place a small amount (approximately 0.1mg) of sample on a piece of PDMAB paper.
2. Press the sample onto the paper with a spatula.

### **Application of Procedure on Evidence (continued):**

3. Place a drop of methanol on the sample spot and allow the methanol to dry.
4. Add hydrochloric acid to the filter paper.
5. Observe the color formation.
6. In some cases, (ie. LSD), the test material is dissolved in solvent, then added to the PDMAB paper. A drop of concentrated hydrochloric acid is added to the paper to develop the color.
7. Record results.

### **Safety Concerns:**

Always wear eye protection, gloves and a laboratory coat when preparing this reagent.

Eye protection and a laboratory coat should be worn when using this reagent for color tests.

Concentrated hydrochloric acid is a strong oxidizing agent and corrosive.

### **Literature References:**

Moffat, A. C., ed., **Clarke's Isolation and Identification of Drugs**, 2nd Edition, Pharmaceutical Press, London, 1986, p. 132.

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Johns, S.H., "Spot Tests: A Color Chart Reference for Forensic Chemists", **Journal of Forensic Science**, July, 1979, pp. 631-649.

This procedure has been used in the Drug Chemistry Section since 1971.

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Procedure A-08	Preliminary Tests Froehde's Reagent	
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**Name of Procedure:**

Preliminary Tests  
Froehde's Reagent

**Suggested Uses:**

The Froehde's reagent consists of a solution of molybdic acid and concentrated sulfuric acid. Aromatic compounds that typically undergo oxidation/reduction/substitution reactions will react with this reagent to produce colored intermediates. Refer to pages 631-649, "Spot Tests: A Color Chart Reference for Forensic Chemists", (see **Literature References**) for color formations of various drugs.

**Apparatus Needed to Perform Procedure Including Preparation of Reagent:**

Fume hood  
Gloves  
Eye protection  
Laboratory coat  
Pipet with bulb  
Graduated cylinder  
50ml beaker  
Glass stirring rod  
Sulfuric acid (concentrated)  
Molybdic acid  
Funnel  
Reagent bottle  
Porcelain spot plate  
Spatula  
Hot plate

**Formula for Preparing Reagent:**

1. Measure out 10 milliliters of concentrated sulfuric acid and heat.



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2. Dissolve 50 milligrams of molybdic acid (or sodium molybdate) in sulfuric acid with heating and stirring.

**Formula for Preparing Reagent (Continued):**

3. When cooled, pour solution into a reagent bottle.
4. Properly label reagent bottle.

**Quality Control:**

A quality control check of this reagent will be performed using a known standard of heroin and following the application procedure listed below.

**Expiration Date of Chemical Reagent:**

The Froehde's reagent should be prepared every 30 days.

**Application of Procedure on Evidence:**

1. Place 1-2 drops of the reagent into a clean well on a spot plate.
2. With a spatula, add approximately 0.1 milligram of the unknown powder/tablet to the reagent in the spot plate.
3. Observe color produced.
4. Record results.

**Safety Concerns:**

Always wear eye protection, gloves and a laboratory coat when preparing this reagent.

Eye protection and a laboratory coat should be worn when using this reagent for color tests.

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Sulfuric acid is a strong oxidizing agent and corrosive.

**Literature References:**

Butler, William P., **Methods of Analysis**, IRS Publication #341, 1966, p. 136.

Johns, S.H., "Spot Tests: A Color Chart Reference for Forensic Chemists", **Journal of Forensic Science**, July, 1979, pp. 631-649.

This procedure has been used in the Drug Chemistry Section since 1971.

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Procedure A-09	Preliminary Tests Mecke's Reagent	
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**Name of Procedure:**

Preliminary Tests  
Mecke's Reagent

**Suggested Uses:**

The Mecke Color test consists of a solution of selenious acid and concentrated sulfuric acid. Aromatic compounds that typically undergo oxidation/reduction/substitution reactions will react with this reagent to produce colored intermediates. Refer to pages 631-649, "Spot Tests: A Color Chart Reference for Forensic Chemists", (see **Literature References**) for color formations of various drugs.

**Apparatus Needed to Perform Procedure Including Preparation of Reagent:**

Fume hood  
Gloves  
Eye protection  
Laboratory coat  
Pipet with bulb  
Graduated cylinder  
50ml beaker  
Glass stirring rod  
Sulfuric acid (concentrated)  
Selenious acid  
Funnel  
Reagent bottle  
Porcelain spot plate  
Spatula

**Formula for Preparing Reagent:**

1. Weigh out 0.25 gram of selenious acid.
2. Dissolve in 25 milliliters of concentrated sulfuric acid.

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### **Formula for Preparing Reagent (Continued):**

3. Pour solution into a reagent bottle.
4. Properly label reagent bottle.

### **Quality Control:**

A quality control check of this reagent will be performed using a known standard of heroin and following the application procedure listed below.

### **Expiration Date of Chemical Reagent:**

The Mecke's reagent should be prepared every 30 days.

### **Application of Procedure on Evidence:**

1. Place 1-2 drops of the reagent into a clean well on a spot plate.
2. With a spatula, add approximately 0.1 milligram of the unknown powder/tablet to the reagent in the spot plate.
3. Observe 1-2 minutes for color to be produced.
4. Record results.

### **Safety Concerns:**

Always wear eye protection, gloves and a laboratory coat when preparing this reagent.

Eye protection and a laboratory coat should be worn when using this reagent for color tests.

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Sulfuric acid is a strong oxidizing agent and corrosive.

**Literature References:**

Butler, William P., **Methods of Analysis**, IRS Publication #341, 1966, p. 136.

Johns, S.H., "Spot Tests: A Color Chart Reference for Forensic Chemists", **Journal of Forensic Science**, July, 1979, pp. 631-649.

This procedure has been used in the Drug Chemistry Section since 1971.

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Procedure A-10	Preliminary Tests Silver Nitrate Reagent	
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**Name of Procedure:**

Preliminary Tests  
Silver Nitrate Reagent

**Suggested Uses:**

The Silver Nitrate reagent consists of a solution of silver nitrate and water. This reagent forms a precipitate with Halide ions.

**Apparatus Needed to Perform Procedure Including Preparation of Reagent:**

Fume hood  
Gloves  
Eye protection  
Laboratory coat  
Pipet with bulb  
Graduated cylinder  
50ml beaker  
Glass stirring rod  
Silver Nitrate  
Water  
Funnel  
Reagent bottle  
Porcelain spot plate  
Spatula  
Culture tube (6 X 50mm)

**Formula for Preparing Reagent:**

1. Weigh out 1 gram of silver nitrate.
2. Dissolve in 20 milliliters of water.
3. Pour solution into a reagent bottle.

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4. Properly label reagent bottle.

### **Quality Control:**

A quality control check of this reagent will be performed using a known standard of sodium chloride and following the application procedure listed below.

### **Expiration Date of Chemical Reagent:**

No expiration date. Reagents need to be properly contained in a sealed container and stored in a cool place.

### **Application of Procedure on Evidence:**

1. Place approximately 0.1 milligram sample in a culture tube (6 X 50mm) with a spatula.
2. Dissolve the sample in 1-2 drops of distilled water.
3. Add 1-2 drops of silver nitrate solution.
4. Observe for the formation of a precipitate.
5. Record results.

### **Safety Concerns:**

Always wear eye protection and laboratory coat when preparing this reagent. A laboratory coat should be worn when using this reagent for color tests.

### **Literature References:**

Butler, William P., **Methods of Analysis**, IRS Publication #341, 1966, p. 137.

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Procedure A-11	Preliminary Tests Zwikker Reagent	
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**Name of Procedure:**

Preliminary Tests  
Zwikker Reagent

**Suggested Uses:**

The Zwikker reagent consists of a cupric sulfate solution and a pyridine/chloroform solution. Barbiturates will produce a purple color formation that is transferred to the organic layer.

**Apparatus Needed to Perform Procedure Including Preparation of Reagent:**

Fume hood  
Gloves  
Eye protection  
Laboratory coat  
Pipet with bulb  
Graduated cylinder  
50ml beaker  
Glass stirring rod  
Cupric Sulfate  
Water  
Pyridine  
Chloroform  
Funnel  
Reagent bottles  
Porcelain spot plate  
Spatula  
Culture tubes (6 X 50mm)

**Formula for Preparing Reagent:**

1. Weigh out 0.12 gram of Cupric Sulfate.
2. Dissolve cupric sulfate in 25 milliliters of water.



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3. Pour solution into a reagent bottle.

**Formula for Preparing Reagent (continued):**

4. Properly label reagent bottle.
5. Measure out 1 milliliter of pyridine and add 24 milliliters of chloroform.
6. Pour reagent into a second reagent bottle.
7. Properly label reagent bottle.

**Quality Control:**

A quality control check of this reagent will be performed using a known standard of a barbiturate and following the application procedure listed below.

**Expiration Date of Chemical Reagent:**

No expiration date. Reagents need to be properly contained in a sealed container and stored in a cool place.

**Application of Procedure on Evidence:**

1. Using a spatula, place approximately 0.1 milligram of sample into a culture tube.
2. Add 1 drop of 0.5% cupric sulfate solution.
3. Add 1 drop of 5% pyridine in chloroform solution.
4. Observe for color formation.
5. Record results.

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### **Safety Concerns:**

Always wear eye protection, gloves and a laboratory coat when preparing this reagent. Eye protection and a laboratory coat should be worn when using this reagent. Pyridine is an irritant and a potential health hazard.

### **Literature References:**

Butler, William P., **Methods of Analysis**, IRS Publication #341, December 1966, p. 107.

This procedure has been used in the Drug Chemistry Section since 1971.

DRUG CHEMISTRY SECTION TECHNICAL PROCEDURE MANUAL		
Procedure A-12	Preliminary Tests Barium Chloride Reagent	
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**Name of Procedure:**

Preliminary Tests  
Barium Chloride Reagent

**Suggested Uses:**

The barium chloride reagent consists of a solution of barium chloride in water. This reagent forms a white precipitate of insoluble barium sulfate with sulfate ions.

**Apparatus Needed to Perform Procedure Including Preparation of Reagent:**

Fume hood  
Gloves  
Eye protection  
Laboratory coat  
Pipet with bulb  
Graduated cylinder  
50ml beaker  
Glass stirring rod  
Barium Chloride  
Water  
Funnel  
Reagent bottle  
Porcelain spot plate  
Spatula

**Formula for Preparing Reagent:**

1. Weigh out 3.0 grams of barium chloride.
2. Dissolve in 27.0 milliliters of water.
3. Pour solution into a reagent bottle.
4. Properly label reagent bottle.

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Procedure A-12	Preliminary Tests Barium Chloride Reagent	
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### **Quality Control:**

A quality control check of this reagent will be performed using a known standard of sodium sulfate and following the application procedure listed below.

### **Expiration Date of Chemical Reagent:**

No expiration date. Reagents need to be properly contained in a sealed container and stored in a cool place.

### **Application of Procedure on Evidence:**

1. Place approximately 0.1 milligram sample in a culture tube (6 X 50mm) with a spatula.
2. Dissolve the sample in distilled water.
3. Add 1-2 drops of barium chloride solution.
4. Observe for formation of a precipitate.
5. Record results.

### **Safety Concerns:**

Always wear eye protection and laboratory coat when preparing this reagent. A laboratory coat should be worn when using this reagent.

### **Literature References:**

Moffat, A. C., ed., **Clarke's Isolation and Identification of Drugs**, 2nd Ed.,

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Pharmaceutical Press, London, 1986

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DRUG CHEMISTRY SECTION TECHNICAL PROCEDURE MANUAL		
Procedure A-13	Preliminary Tests Methanolic Potassium Hydroxide Reagent	
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**Name of Procedure:**

Preliminary Tests  
Methanolic Potassium Hydroxide Reagent

**Suggested Uses:**

This reagent is a useful preliminary test for cocaine. Cocaine will react with this reagent to form methyl benzoate, a compound with the characteristic odor of oil of wintergreen.

**Apparatus Needed to Perform Procedure Including Preparation of Reagent:**

Fume hood  
Gloves  
Eye protection  
Laboratory coat  
Pipet with bulb  
Graduated cylinder  
250ml beaker  
Glass stirring rod  
Potassium hydroxide  
Methanol  
Funnel  
Reagent bottle  
Porcelain spot plate  
Spatula

**Formula for Preparing Reagent:**

1. Weigh out 5 grams of potassium hydroxide into a beaker.
2. Add 100 milliliters of methanol and stir until dissolved.
3. Pour solution into reagent bottle.
4. Properly label reagent bottle.

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Procedure A-13	Preliminary Tests Methanolic Potassium Hydroxide Reagent	
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### **Quality Control:**

A quality control check of this reagent will be performed using a known standard of cocaine and following the application procedure listed below.

### **Expiration Date of Chemical Reagent:**

No expiration date. Reagents need to be properly contained in a sealed container and stored in a cool place.

### **Application of Procedure on Evidence:**

1. Place 1-2 drops of the reagent into a clean well on a spot plate.
2. With a spatula, add approximately 0.1 milligram of the unknown powder to the reagent in the spot plate.
3. Carefully sniff to detect if the wintergreen odor is present.
4. Record results.

### **Safety Concerns:**

Always wear eye protection, gloves and a laboratory coat when preparing this reagent.

Eye protection and a laboratory coat should be worn when using this reagent for color tests.

Potassium hydroxide is a strong caustic and may cause severe chemical burns.

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Procedure A-13	Preliminary Tests Methanolic Potassium Hydroxide Reagent	
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**Literature References:**

Jungreis, Ervin, **Spot Test Analysis**, John Wiley & Sons, 1985, p. 80.

This procedure has been used in the Drug Chemistry Section since 1987.



DRUG CHEMISTRY SECTION TECHNICAL PROCEDURE MANUAL		
Procedure A-14	Preliminary Tests Secondary Amine Reagent #1	
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**Name of Procedure:**

Preliminary Tests  
Secondary Amine Reagent #1

**Suggested Uses:**

The secondary amine reagent consists of cupric sulfate, concentrated ammonium hydroxide and toluene-carbon disulfide. This reagent gives a yellow/brown discoloration in the organic phase when a secondary amine is present.

**Apparatus Needed to Perform Procedure Including Preparation of Reagent:**

Fume hood  
Gloves  
Eye protection  
Laboratory coat  
Pipet with bulb  
Graduated cylinder  
50ml beaker  
Glass stirring rod  
Ammonium hydroxide  
Cupric sulfate  
Water  
Toluene  
Carbon Disulfide  
Reagent bottles  
Funnel  
Porcelain spot plate  
Spatula  
Culture tube (6 X 50mm)

**Formula for Preparing Reagent:**

1. Weigh out 0.12 gram of cupric sulfate.

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Procedure A-14	Preliminary Tests Secondary Amine Reagent #1	
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2. Dissolve in 25 milliliters water.
3. Pour solution into a reagent bottle.

**Formula for Preparing Reagent (continued):**

4. Properly label reagent bottle.
5. Measure out 25 milliliters of concentrated ammonium hydroxide.
6. Pour into a separate reagent bottle.
7. Properly label reagent bottle.
8. Measure out 20 milliliters of toluene and mix with 5 milliliters of carbon disulfide.
9. Pour into a separate reagent bottle.
10. Properly label reagent bottle.

**Quality Control:**

A quality control check of this reagent will be performed using a known standard of methamphetamine and following the application procedure listed below.

**Expiration Date of Chemical Reagent:**

No expiration date. Reagents need to be properly contained in a sealed container and stored in a cool place.

**Application of Procedure on Evidence:**

1. With spatula, place 0.1 milligram of the sample in a culture tube.
2. Add 2-3 drops of the cupric sulfate solution to dissolve sample.

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3. Add 2-3 drops of the concentrated ammonium hydroxide.
4. Add 2-3 drops of the toluene/carbon disulfide solution.
5. Agitate culture tube.
6. Observe for a yellow/brown discoloration in the organic phase.
7. Record results.

**Safety Concerns:**

Always wear eye protection and laboratory coat when preparing this reagent. A laboratory coat should be worn when using this reagent for color tests.

**Literature References:**

Procedure used in Drug Chemistry Section since 1971.

DRUG CHEMISTRY SECTION TECHNICAL PROCEDURE MANUAL		
Procedure A-15	Preliminary Tests Secondary Amine Reagent #2	
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**Name of Procedure:**

Preliminary Tests  
Secondary Amine Reagent #2

**Suggested Uses:**

This is a color test used to distinguish methamphetamine from amphetamine. Methamphetamine and other secondary amines form blue-violet compounds with this reagent, while amphetamine and other primary amines yield a slow pink to cherry red color.

**Apparatus Needed to Perform Procedure Including Preparation of Reagent:**

Fume hood  
Gloves  
Eye protection  
Laboratory coat  
Pipet with bulb  
Graduated cylinder  
Beakers  
Glass stirring rod  
Sodium nitroprusside  
Acetaldehyde  
Sodium carbonate  
Funnel  
Reagent bottles  
Porcelain spot plate  
Spatula  
Culture tubes (6 X 50mm)

**Formula for Preparing Reagent:**

**Reagent A:**

1. Dissolve 1 gram of sodium nitroprusside in 100 milliliters of water.

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- To 90 milliliters of this solution, add 10 milliliters of acetaldehyde.
- Pour solution into a reagent bottle.

**Formula for Preparing Reagent (continued):**

- Properly label reagent bottle.

**Reagent B:**

- Dissolve 2 grams of sodium carbonate in 100 milliliters of water.
- Pour solution into a reagent bottle.
- Properly label reagent bottle.

**Quality Control:**

A quality control check of this reagent will be performed using a known standard of methamphetamine and following the application procedure listed below.

**Expiration Date of Chemical Reagent:**

Reagent A must be prepared monthly and refrigerated.

Reagent B has no expiration date. Reagent B needs to be properly contained in a sealed container and stored in a cool place.

**Application of Procedure on Evidence:**

- With a spatula, add a small amount of the suspected material to a culture tube or clean well on a spot plate.
- Add 1 drop of Reagent A, then 2 drops of Reagent B.

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Procedure A-15	Preliminary Tests Secondary Amine Reagent #2	
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3. Observe the color change.
4. Record results.

**Safety Concerns:**

Always wear eye protection, gloves and a laboratory coat when preparing this reagent.

Eye protection and a laboratory coat should be worn when using this reagent for color tests.

**Literature References:**

Feigl, Fritz, **Spot Tests in Organic Analysis**, Elsevier Publishing Co., 1956, p. 260.

DRUG CHEMISTRY SECTION TECHNICAL PROCEDURE MANUAL		
Procedure A-16	Preliminary Tests Liebermann's Reagent	
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**Name of Procedure:**

Preliminary Tests  
Liebermann's Reagent

**Suggested Uses:**

Liebermann's Reagent consists of a solution of sodium or potassium nitrite dissolved in sulfuric acid. The test was originally developed to give intense colors with phenols. Orange colors are produced by certain substances that contain a monosubstituted benzene ring. Orange or brown colors are produced by some substances that contain two monosubstituted benzene rings. Other colors produced may be found in the reference listed below.

**Apparatus Needed to Perform Procedure Including Preparation of Reagent:**

Fume hood  
Gloves  
Eye protection  
Laboratory coat  
Balance  
Graduated cylinder  
Beaker of ice (ice bath)  
Glass stirring rod  
Sulfuric Acid (concentrated)  
Sodium or Potassium Nitrite  
Reagent bottles  
Porcelain spot plate  
Spatula

**Formula for Preparing Reagent:**

1. Measure out 10 milliliters of concentrated sulfuric acid in a graduated cylinder.
2. Place the graduated cylinder with sulfuric acid into an ice bath.
3. Measure out 1 gram of sodium or potassium nitrite.
4. Slowly add the sodium or potassium nitrite to the sulfuric acid.
5. Keep cooling the reagent and swirl to absorb the brown fumes.

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Procedure A-16	Preliminary Tests Liebermann's Reagent	
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6. Let cool, and pour reagent into the reagent bottle.
7. Properly label the reagent bottle.

### **Quality Control:**

A quality control check of this reagent will be performed using a known standard of methamphetamine and following the application procedure listed below.

### **Expiration Date of Chemical Reagent:**

The Liebermann's Reagent should be prepared every six months.

### **Application of Procedure on Evidence:**

1. Place 1-2 drops of the reagent into a clean well on a spot plate.
2. With a spatula, add approximately 0.1 milligram of the unknown substance to the reagent in the spot plate.
3. Observe color produced.
4. Record results.

### **Safety Concerns:**

Always wear eye protection, gloves and a laboratory coat when preparing this reagent.

Sulfuric acid is a strong oxidizing agent and corrosive.

Sodium and potassium nitrite are strong oxidizing agents and may react violently with sulfuric acid if added too rapidly.

### **Literature References:**

1. Moffat, A.C., Osselton, M.D., and Widdop, B., Clarke's Analysis of Drugs and Poisons, Pharmaceutical Press, London, 2004, pp.287-288.



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DRUG CHEMISTRY SECTION TECHNICAL PROCEDURE MANUAL		
Procedure A-17	Preliminary Tests Ketamine Color Test	
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**Name of Procedure:**

Preliminary Tests  
Ketamine Color Test

**Suggested Uses:**

This is a color test used specifically for Ketamine. The color, color change, and time frames constitutes a highly specific screening test for ketamine. The test is negative for amphetamine, methamphetamine, MDA, MDMA, and Phencyclidine (PCP), all of which are occasionally encountered with ketamine.

**Apparatus Needed to Perform Procedure Including Preparation of Reagent:**

Fume hood  
Gloves  
Eye protection  
Laboratory coat  
Pipet with bulb  
Graduated cylinder  
Beakers  
Glass stirring rod  
Gold Bromide  
Deionized water  
Sodium Hydroxide  
Funnel  
Reagent bottles  
Porcelain spot plate  
Spatula  
Culture tubes (6 X 50mm)

**Formula for Preparing Reagent:**

**Reagent A: 0.5% Gold Bromide (50mL):**

Dissolve 0.25 grams of Gold Bromide in 50 milliliters of deionized water.

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**Reagent B: 0.2M solution of NaOH (10mL):**

Dissolve 0.08 grams of Sodium hydroxide in 10 milliliters of deionized water.

**Quality Control:**

A quality control check of this reagent will be performed using a known standard of ketamine and following the application procedure listed below.

**Expiration Date of Chemical Reagent:**

Reagents A and B have expirations of 3 months and should be refrigerated.

**Application of Procedure on Evidence:**

1. In a ceramic spot plate, place one drop of the 0.5% Gold Bromide solution.
2. Add 1 drop of sample to the same well.
3. Add 1 drop of the 0.2M NaOH solution to the well.
4. After one minute, Ketamine gives a purple color. After two minutes, the color changes to a dark, purple-black. If an instantaneous color change occurs, the test cannot be used for presumptive identification of ketamine.

**Safety Concerns:**

Always wear eye protection, gloves and a laboratory coat when preparing this reagent.

Eye protection and a laboratory coat should be worn when using this reagent for color tests.

DRUG CHEMISTRY SECTION TECHNICAL PROCEDURE MANUAL		
Procedure A-17	Preliminary Tests Ketamine Color Test	
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**Literature References:**

1. Mohammad Sarwar, Ph.D-DEA Microgram Journal, Volume 4, Numbers 1-4 (January-December 2006)

DRUG CHEMISTRY SECTION TECHNICAL PROCEDURE MANUAL		
Procedure A-18	Preliminary Tests Modified Cobalt Thiocyanate Test	
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**Name of Procedure:**

Preliminary Tests  
Modified Cobalt Thiocyanate Test

**Suggested Uses:**

The Modified Cobalt Thiocyanate test consists of an aqueous solution of Cobalt thiocyanate (Procedure A-03, Cobalt Thiocyanate Reagent), concentrated hydrochloric acid, and chloroform. This test reacts with the base (non water soluble) form of some secondary and tertiary amines and other alkaloids to produce a blue color. After addition of chloroform, the blue color is transferred to the chloroform phase.

**Apparatus Needed to Perform Procedure Including Preparation of Reagent:**

Gloves  
Eye protection  
Laboratory coat  
Cobalt thiocyanate Reagent (Procedure A-03)  
Concentrated hydrochloric acid  
Chloroform  
Reagent bottles  
Porcelain spot plate  
Spatula

**Formula for Preparing Reagent:**

1. Follow Procedure A-03 for the preparation of the Cobalt Thiocyanate Reagent and properly label reagent bottle.

**Quality Control Check:**

A quality control check of this reagent will be performed using a known standard of cocaine and following the application procedure listed below.

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### **Expiration Date of Chemical Reagent:**

No expiration date. Reagents need to be properly contained in a sealed container and stored in a cool place.

### **Application of Procedure on Evidence:**

1. Place 1-2 drops of the Cobalt Thiocyanate Reagent into a clean well of a spot plate.
2. With a spatula, add approximately 0.1 milligram of the unknown powder/tablet to the reagent in the spot plate.
3. Add 1 drop of concentrated hydrochloric acid and observe the color change.
4. Add 2-3 drops of chloroform.
5. Allow phases to separate and observe the color in the bottom chloroform layer. A blue color with the acid addition, and a blue color transfer to the chloroform layer is indicative of a positive test.
6. Record results.

### **Safety Concerns:**

Always wear eye protection and laboratory coat when preparing this reagent. A laboratory coat should be worn when using this reagent for color tests.

### **Literature References:**

Johns, S.H., "Spot Tests: A Color Chart Reference for Forensic Chemists", **Journal of Forensic Science**, July, 1979, pp. 631-649.

Deakin, A.L., "A Study of Acids used for the Acidified Cobalt Thiocyanate Test for Cocaine Base", **Microgram Journal Technical Note**, Volume 1, Number 1-2, Jan-June 2003, pp. 40-43.

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Smith, F.P, Siegal, J.A., Athanaselis, S.A., "Cocaine: Methods of Forensic Analysis", **Handbook of Forensic Drug Analysis**, pp. 240-242.

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